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Environmental Technology Verification Program

Safe Buildings Monitoring and Detection Technology Verification Program

Test/QA Plan for Verification of Portable Ion Mobility Spectrometers for Detection of Chemicals and Chemical Agents in Buildings

Test/QA Plan

for

VERIFICATION OF PORTABLE ION MOBILITY SPECTROMETERS FOR DETECTION OF CHEMICALS AND CHEMICAL AGENTS IN BUILDINGS

Version 2
June 17, 2004

Signed by Eric Koglin	**July 1, 2004**
EPA TASK ORDER PROJECT OFFICER	**Date**
Signed by George Brilis	**July 8, 2004**
EPA QUALITY MANAGER	**Date**
Signed by Karen Riggs	**June 28, 2004**
BATTELLE PROGRAM MANAGER	**Date**
Signed by Zachary Willenberg	**June 17, 2004**
BATTELLE QUALITY MANAGER	**Date**

Battelle
505 King Avenue
Columbus, OH 43201-2693

TEST/QA PLAN

for

VERIFICATION OF PORTABLE ION MOBILITY SPECTROMETERS FOR DETECTION OF CHEMICALS AND CHEMICAL AGENTS IN BUILDINGS

Prepared by

Battelle
Columbus, Ohio

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EPA Task Order Project Officer
Eric Koglin

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LIST OF ACRONYMS

AC	hydrogen cyanide
APT	Aerosol and Process Technologies
AS	Atmospheric Sciences
CET	Chemical and Environmental Technologies
CG	phosgene
CK	cyanogen chloride
Cl_2	chlorine
CRDEC	Chemical Research, Development and Engineering Center
CSM	chemical surety material
CW	chemical warfare
CWA	chemical warfare agent
DEAE	N,N-diethylaminoethanol
DOD	Department of Defense
DOE	Department of Energy
EC	electrochemical
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
FID	flame ionization detector
FPD	flame photometric detector
ft	foot
FTIR	Fourier transform infrared
GB	sarin
GC	gas chromatography
GD	soman
HD	sulfur mustard
HML	Hazardous Materials Laboratory
HMRC	Hazardous Materials Research Center
IDLH	Immediately Dangerous to Life and Health
IMS	Ion mobility spectrometer
L	Lewisite
LITF	Large Item Test Facility
min	minute
MREF	Medical Research and Evaluation Facility
MSD	mass selective detector
NHSRC	National Homeland Security Research Center
PE	performance evaluation
PPE	personal protective equipment
ppm	parts per million
QA	quality assurance
QC	quality control
QMP	quality management plan

LIST OF ACRONYMS (Continued)

RDS	research dilute solution
RH	relative humidity
RSD	relative standard deviation
SA	arsine (AsH_3)
SBCCOM	U.S. Army Soldier Biological and Chemical Command
SOP	standard operating procedure
TIC	toxic industrial chemical
TOPO	task order project officer
TSA	technical systems audit
Y/N	yes/no

DISTRIBUTION LIST

Dr. Thomas J. Kelly
Battelle
505 King Avenue
Columbus, Ohio 43201-2693

Ms. Karen Riggs
Battelle
505 King Avenue
Columbus, Ohio 43201-2693

Mr. Zachary Willenberg
Battelle
505 King Avenue
Columbus, Ohio 43201-2693

Mr. Kent Hofacre
Battelle
505 King Avenue
Columbus, Ohio 43201-2693

Mr. Dale Folsom
Battelle
505 King Avenue
Columbus, Ohio 43201-2693

Ms. Tricia Derringer
Battelle
505 King Avenue
Columbus, Ohio 43201-2693

Mr. Eric Koglin
USEPA National Homeland Security
Research Center
944 East Harmon Avenue
Las Vegas, NV 89119

Technology Vendor(s)

Vendor Approval of EPA/ETV Test/QA Plan for

VERIFICATION OF PORTABLE ION MOBILITY SPECTROMETERS FOR DETECTION OF CHEMICALS AND CHEMICAL AGENTS IN BUILDINGS

Version 2
June 17, 2004

Name _____

Signature _____

Company _____

Date _____

1.0 INTRODUCTION

1.1 Test Description

The U.S. Environmental Protection Agency (EPA) has the responsibility to help protect the public in workplaces and other buildings that may be subject to attack using chemical or biological agents. That responsibility includes identifying methods and equipment for detecting or monitoring for chemical and biological contaminants in indoor environments. In January 2003, EPA established the National Homeland Security Research Center (NHSRC) to manage, coordinate, and support a wide variety of homeland security research and technical assistance efforts. Through the Safe Buildings Program, a key research component of the NHSRC, EPA is verifying the performance of products, methods, and equipment that can detect chemical or biological agents on indoor surfaces or in indoor air. EPA's goal is to generate objective performance data so building and facility managers, first responders, and other technology buyers and users can make informed purchase and application decisions.

To meet this goal, EPA is using the process established in its Environmental Technology Verification (ETV) Program. The ETV process, which has been used since 1997 to verify the performance of over 200 environmental technologies, includes developing a test/quality assurance (QA) plan (with input from stakeholders and vendors), applying high-quality test procedures according to that plan, and publicizing separate performance reports for each technology verified. The purpose of ETV is to provide objective and quality-assured performance data on environmental technologies, so that users, developers, regulators, and consultants have an independent and credible assessment of what they are buying and recommending. The ETV process does not rank, select, or approve technologies, but instead provides credible performance data to potential users and buyers. Other information about the program is available at the ETV web site (http://www.epa.gov/etv) and through the NHSRC web site (www.epa.gov/nhsrc).

This test/QA plan provides procedures for verification of commercially available portable ion mobility spectrometer (IMS) detectors that can rapidly detect individual chemicals and

chemical agents in indoor air. The verification test will be conducted in accordance with the ETV process and will be conducted by Battelle, of Columbus, OH, under the direction of the EPA. In performing this verification test, Battelle will follow the procedures specified in this test/QA plan and will comply with quality requirements in the Quality Management Plan (QMP) for the ETV Safe Buildings Monitoring and Detection Technology Verification Program.[1]

1.2 Test Objective

The objective of the verification test is to assess the performance of commercial portable IMS technologies by challenging them with a variety of toxic industrial chemicals (TICs), and chemical warfare (CW) agents, under a range of conditions and practices that mimic the real-world use of these instruments. This verification is focused on the use of portable IMS instruments by first responders to identify contaminants and guide emergency response activities after chemical contamination of a building. The performance characteristics to be evaluated include the ability to detect and identify target agents and chemicals under both ideal and realistic operating conditions. The response time, response threshold, accuracy, recovery time, temperature and humidity effects, interference effects, and battery life of the instruments will be assessed. Operational factors such as cold/hot start behavior, cost, ease of use, and data output capability will also be evaluated.

1.3 Organization and Responsibilities

The verification test will be performed by Battelle under the direction of EPA, with input from the vendors whose IMS instruments will be verified. The organization chart in Figure 1 shows the individuals from Battelle, the vendor companies, and EPA who will have responsibilities in the verification test. The specific responsibilities of these individuals are detailed in the following paragraphs.

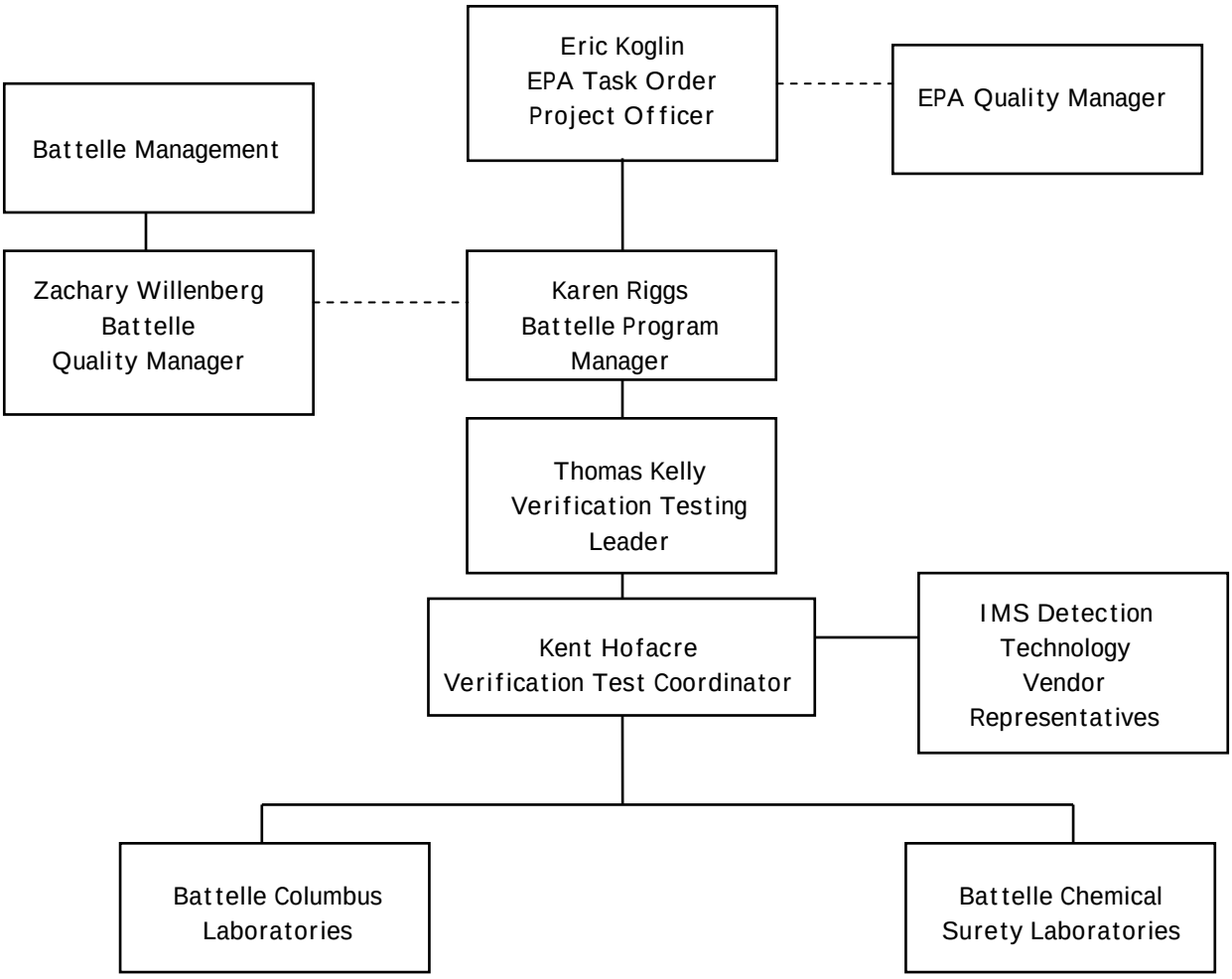

Figure 1. Organization Chart for the IMS Detection Technology Verification Test

1.3.1 Battelle

Mr. Kent Hofacre is Battelle's Verification Test Coordinator for this verification test. In that role, Mr. Hofacre will oversee the verification testing of portable IMS detection technologies. Mr. Hofacre's responsibilities are to:

- Select the appropriate laboratory or location for the test.
- Coordinate with vendor representatives to facilitate the performance of testing.
- Prepare the draft test/QA plan, verification reports, and verification statements.
- Arrange for use of the test facility and establish a test schedule.
- Arrange for the availability of qualified staff to conduct the test.
- Assure that testing is conducted according to this test/QA plan.
- Revise the test/QA plan, verification reports, and verification statements in response to reviewers' comments.
- Keep the Battelle Program Manager and Verification Testing Leader informed of progress and difficulties in planning and conducting the test.
- Coordinate with the Battelle Quality Manager for the performance of technical and performance audits as required by Battelle or EPA Quality Management staff.
- Guide the Battelle/EPA/vendor team in performing the verification test in accordance with this test/QA plan.
- Have overall responsibility for ensuring that this test/QA plan is followed.
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary.
- Establish a budget and schedule for the verification test and direct the effort to ensure that budget and schedule are met.
- Coordinate distribution of final test/QA plan, verification reports, and statements.

Dr. Thomas J. Kelly is the Verification Testing Leader in this program. In this role, Dr. Kelly will support Mr. Hofacre by:

- Ensuring that ETV program procedures are being followed.
- Providing a technical review of the draft test/QA plan, verification reports, and verification statements.
- Serving as backup Verification Test Coordinator in Mr. Hofacre's absence.

Ms. Karen Riggs is Battelle's Program Manager for this program. As such, Ms. Riggs will:

- Maintain communication with EPA's Task Order Project Officer (TOPO) on all aspects of the program.
- Monitor adherence to budgets and schedules in this work.

- Provide the TOPO with monthly technical and financial progress reports.
- Review the draft test/QA plan.
- Review the draft verification reports and statements.
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test.
- Ensure that vendor confidentiality is maintained.
- Support Mr. Hofacre in responding to any issues raised in assessment reports and audits.

Mr. Zachary Willenberg is Battelle's Quality Manager for this program. As such, Mr. Willenberg will:

- Review the draft test/QA plan.
- Maintain communication with EPA Quality Management staff for this program.
- Conduct a technical systems audit (TSA) at least once during the verification test.
- Review results of performance evaluation (PE) audit(s) specified in this test/QA plan.
- Audit at least 10% of the verification data.
- Prepare and distribute an assessment report for each audit.
- Verify implementation of any necessary corrective action.
- Notify Battelle's Program Manager to issue a stop work order if internal audits indicate that data quality is being compromised. Notify the Verification Test Coordinator if such an order is issued.
- Provide a summary of the QA and quality control (QC) activities and results for the verification reports.
- Review the draft verification reports and statements.
- Ensure that all quality procedures specified in this test/QA plan and in the QMP[(1)] are followed.

Battelle technical staff will support Mr. Hofacre in planning and conducting the verification test. These staff will:

- Assist in planning and scheduling the verification test.
- Become familiar with the use of the IMS detection technologies to be tested.
- Carry out the test procedures specified in this test/QA plan.
- Assure that test procedures and data acquisition are conducted according to this test/QA plan.

1.3.2 Vendors

Vendors of portable IMS detection technologies will:

- Provide input for preparation of the draft test/QA plan.
- Review the test/QA plan and approve the current version prior to verification testing of their technology.
- Sign a Vendor Agreement specifying the respective responsibilities of the vendor and of Battelle in the verification test.
- Provide information on the quantitative response of their portable IMS instruments (e.g., programmed alarm levels; concentrations triggering transition between low/medium/high readings) to aid in planning of the verification test.
- Provide at least two units of their portable IMS detection technology for use in the verification test.
- Train Battelle and/or test facility staff in the operation of their portable IMS instruments.
- Provide support, if needed, in use of the IMS instruments during testing.
- Review their respective draft verification report and verification statement.

1.3.3 EPA

Mr. Eric Koglin is EPA's TOPO for this program. As such, Mr. Koglin will:

- Have overall responsibility for directing the verification process.
- Review the draft test/QA plan.
- Approve the final test/QA plan and any subsequent versions.
- Review the draft verification reports and statements.
- Oversee the EPA review process on the draft test/QA plan, reports, and verification statements.
- Coordinate the submission of verification reports and statements for final EPA approval.

The EPA Quality Manager for this program will:

- Review the draft test/QA plan and any subsequent versions.
- Perform, at his/her option, one external TSA during the verification test.
- Notify the EPA TOPO to issue a stop work order if an external audit indicates that data quality is being compromised.
- Prepare and distribute an assessment report summarizing the results of the external audit, if one is performed.
- Review the draft verification reports and statements.

1.3.4 Test Facility

The location for the verification test described here will be Battelle's laboratories in Columbus and West Jefferson, Ohio. The Columbus facilities to be used are chemical laboratories equipped for safe handling of volatile TICs. The West Jefferson facilities are chemical surety laboratories certified for use of CW agents. Other test facilities could be used depending on the availability and capability of the facilities. In general, the responsibilities of the technical staff in these test facilities will be to:

- Ensure that the facility is fully functional prior to the times/dates needed in the verification test.
- Provide requisite technical staff during the verification test.
- Provide any safety training needed by Battelle, vendor, or EPA staff.
- Review and approve all data and records related to facility operation.
- Review the test/QA plan.
- Adhere to the requirements of the test/QA plan and the program QMP[(1)] in carrying out the verification test.
- Provide input on facility procedures for the verification test report.
- Support Mr. Hofacre in responding to any issues raised in assessment reports and audits related to facility operation.

2.0 APPLICABILITY

2.1 Subject

This test/QA plan focuses on the verification testing of commercially available portable IMS detectors for detection of toxic chemicals or chemical agents in indoor air. This plan is specifically focused on detection in the building environment, in the context of use of the IMS instruments by first responders arriving at a potential contamination event. In this target scenario, there is need for immediate and accurate identification of chemicals, by first responders who are wearing extensive personal protective equipment (PPE), regardless of the weather or environmental conditions at the time. These needs are the basis for the test procedures stated in this plan.

The chemicals and chemical agents that may pose a threat in the building environment may include TICs and CW agents. Chemical agents having relatively low vapor pressures are of interest in this test, because of their persistence in the building environment. However, highly volatile TICs and CW agents are also included in testing under this plan; although they can be readily removed from the building by ventilation, they may be present at the time that first responders arrive at the scene.

Verification testing requires a basis for establishing the quantitative performance of the tested technologies. For this verification, quantitative performance is assessed primarily in terms of the detection of the TICs and CW agents. In testing, the TIC and CW agent concentrations sampled by the IMS instruments are confirmed by documented independent methods so that IMS performance can be accurately determined.

2.2 Scope

The overall objective of the test described in this plan is to verify the performance of the portable IMS technologies with selected TICs and CW agents under a realistically broad range of indoor conditions and procedures of use. Testing will be conducted over ranges of temperature

and relative humidity (RH) representing those that might be encountered in an emergency response situation in a building environment. The rigorous nature of actual use by first responders will be simulated by testing for cold and hot start operation, battery life, and interferences. To the extent possible, in all testing two units of each IMS instrument will be tested simultaneously. The test data sets from the two units will be compiled and reported as independent measures of the IMS performance. However, in the event of failure of one of the IMS units during testing, the testing will continue with only one unit until the second unit can be repaired or replaced.

The performance parameters on which the portable IMS instruments will be evaluated under this plan include:

- Response time
- Recovery time
- Accuracy
- Repeatability
- Response threshold
- Temperature and humidity effects
- Interference effects
- Cold/hot start behavior
- Battery life
- Ease of use
- Data output
- Cost.

The response time, recovery time, accuracy, and repeatability will be evaluated by challenging the IMS instruments with known vapor concentrations of TICs and CW agents. Performance of such tests with low target analyte concentrations will evaluate the response threshold of the IMS instruments. Similar tests conducted over a range of temperature and RH will be used to establish the effects of these factors on detection capabilities. The effects of potential interferences in an emergency situation will be assessed, by sampling those interferences both with and without the target TICs and CW agents present. Testing the IMS instruments after a cold start (i.e., without the usual warm-up period) and after hot storage will evaluate the delay time before readings can be obtained, and the response speed and accuracy once readings are obtained. Battery life will be determined as the time until IMS performance

degrades as battery power is exhausted, in continuous operation. Operational factors such as ease of use, data output, and cost will be assessed by observations of the test personnel and through inquiries to the technology vendors.

The testing to be conducted under this plan is limited to detection of chemicals in the vapor phase, because that mode of application is most relevant to the stated target scenario, i.e., use by first responders. It is conceivable that an IMS instrument may be capable of analyzing surface wipe samples, or heating a sample surface to promote vaporization of chemical agents. Such capabilities could be addressed by a modification of this test/QA plan. However, those capabilities are unlikely to be used by first responders at a scene of building contamination, and so are not addressed in this verification. Testing will be conducted in two phases: the first will address detection of TICs, and will be conducted in a non-surety laboratory; the second will address detection of CW agents, and will be conducted in a certified surety facility.

Because of the nature of the test activities under this test/QA plan, the IMS instruments will be operated by Battelle staff in all testing. However, each IMS vendor will be required to provide the appropriate instructions or operator's manuals for their instrument, and to train Battelle staff in the correct use of the instrument. Battelle testing staff will review all written instructions and manuals before receiving training from the vendor. The Battelle testing staff will note the clarity, completeness, and adequacy of the written documentation provided. When each IMS vendor is satisfied that Battelle staff are fully trained in operating the IMS instrument, the vendor will be required to attest in writing that the Battelle staff are authorized to operate the IMS instrument for the purpose of this verification test.

The portable IMS instruments to be tested may provide different types of data outputs that must be addressed under this test/QA plan. Although some IMS instruments may provide quantitative indication of the concentration of the target CW agent or TIC, others may provide only qualitative (e.g., an audible or visual alarm indicating the presence of the compound) or semi-quantitative (low/medium/high reading, numbered bar graph, etc.) indications. To achieve the most effective verification test, the IMS vendors will be asked to provide the nominal concentrations of target compounds that correspond to the qualitative detection ranges, thresholds, or transition points of their IMS instruments. For example, the vendor of an IMS

instrument that provides low/medium/high indications will be asked to provide the nominal concentrations of selected agents and TICs that are programmed to cause a transition in reading from low to medium, and medium to high. These nominal levels will be factored into the test procedures, to assure that relevant information on IMS performance is obtained.

3.0 SITE DESCRIPTION

These tests are expected to be conducted at Battelle facilities in Columbus and West Jefferson, Ohio. Those facilities are described below. Alternative facilities could also be used, provided those facilities meet all the requirements for safety, security, and testing capability established by this plan.

3.1 General Site Description

Battelle has two primary campuses in or near Columbus, Ohio that will be used to conduct the verification tests. The main chemistry laboratories for non-chemical surety material testing are located in a new King Avenue laboratory. Testing with the non-surety material – TICs and interferents – will be conducted in the King Avenue laboratory. These facilities have the dedicated vapor generation, collection, and analysis equipment needed to conduct the tests described in this plan. The King Avenue laboratory has been used previously to conduct instrument and filter tests using phosgene (CG), hydrogen cyanide (AC), cyanogen chloride (CK), and chlorine (Cl_2) under controlled environmental conditions.

Battelle's West Jefferson facility is an 1,800-acre research campus located within a tract of Battelle-owned land in a rural area approximately 17 miles west of downtown Columbus, Ohio. Testing with CW agents under this test/QA plan will use either the Medical Research and Evaluation Facility (MREF) or the Hazardous Materials Research Center (HMRC) at West Jefferson, both of which conduct research with chemical surety material (CSM).

Battelle's Medical Research and Evaluation Facility (MREF) is a Department of Defense laboratory-scale facility conducting research with chemical and biological agents. The MREF is licensed to ship, receive, and handle select agents, as defined by the Centers for Disease Control and Prevention. The facility maintains state-of-the-art equipment and professional and technical staffing expertise to safely conduct testing and evaluation of hazardous chemical and biological materials.

The MREF and its personnel have capability for storing and safely handling CW agents. Handling of CW agents at the MREF is detailed in the following standard operating procedures (SOP): MREF SOP I-002 *Storage, Dilution, and Transfer of GA, GB, GD, GF, TGD, VX, HD, HL, HN and L when CA Concentration/Quantity is Greater than Research Dilute Solution (RDS)*, MREF SOP I-003 *Receipt, Transfer, Storage, and Use of Research Dilute Solution (RDS)*, and MREF SOP I-004 *Disposal of Chemical Agent*.

Battelle's HMRC is an ISO 9001 certified facility that provides a broad range of materials testing, system and component evaluation, research and development, and analytical chemistry services that require the safe use and storage of highly toxic substances. Since its initial certification by the Chemical Research, Development and Engineering Center in 1981, the facility has functioned as both a research and a technology development laboratory in support of DoD chemical programs. The HMRC can safely store and handle 3-quinuclidinyl benzilate (BZ), tabun (GA), sarin (GB), soman (GD), thickened GD (TGD), sulfur mustard (HD), thickened HD (THD), Lewisite (L), mustard-Lewisite mixtures (HL), V-agent (VX), and other hazardous materials and toxins, such as arsine (AsH_3; SA), cyanogen chloride (CK), hydrogen cyanide (AC), phosgene (CG), perfluoroisobutylene (PFIB), as well as agent simulants, Class A poisons, and toxins (e.g., T-2 toxin). Determination of delivered CW agent concentrations in this work will be conducted according to HMRC SOP *"HMRC-IV-056-07 for Operation and Maintenance of Gas Chromatographs and for the Analysis of Solutions Containing GA, GB, GD, GF, HD, and VX by Gas Chromatography."*

The HMRC complex consists of approximately 10,000 ft^2 which includes the Hazardous Materials Laboratory and the Large Item Test Facility (LITF), which provide approximately 2,000 ft^2 of laboratory space and 100 linear ft of CSM-approved filtered hoods for working with neat (pure) CSM; about 630 ft^2 of research dilute solution (RDS, i.e., diluted chemical agent) laboratory space, including four fume hoods; and approximately 2,100 ft^2 of laboratory support areas, including environmental monitoring, emergency power supplies, and air filter systems. The LITF, which occupies approximately 540 ft^2 of the HMRC, was designed and is operated for test and evaluation of items and systems too large to fit into standard laboratory fume hoods.

3.2 Site Operations

Battelle operates its certified chemical surety facilities in compliance with all applicable Federal, state, and local laws and regulations, including Army Regulations. Battelle's facilities are certified through inspection by personnel from the appropriate government agency. Battelle is certified to work with CSM through its Bailment Agreement DAAD13-H-03-0003 with the U.S. Army Research, Development & Engineering Command (RDECOM). RDECOM officials and the Army Material Command Inspector General for Chemical Surety Sites regularly inspect Battelle's facilities to ensure that Battelle continues to operate its chemical surety laboratories in accordance with all applicable federal regulations. Additionally, the HMRC is ISO 9001 certified, performs work under this ISO standard, and is monitored by regular outside ISO quality inspections. Our chemical agent facilities and attendant certifications are listed in Table 1. Battelle's agent stocks will be analyzed prior to testing to verify the purity of the agent used to make the test samples. Only chemical agents (CA) with purity greater than 85 percent will be used in this program.

Table 1. Battelle Facilities for CW Agent Testing of Portable IMS Instruments

Facility	Materials	Level	Certification
Medical Research and Evaluation Facility	CW Agents	Chemical Surety Materiel (CSM) (Neat) RDT & E (Dilute)	United States of America Medical Research Materiel Command (USAMRMC) No. G472501
Hazardous Materials Research Center	CW Agents	Chemical Surety Materiel (CSM) (Neat) RDT & E (Dilute)	Bailment Agreement No. DAAD13-H-03-0003
Analytical Chemistry Laboratory	CW Agents	RDT & E (Dilute)	Bailment Agreement No. DAAD13-H-03-0003

4.0 EXPERIMENTAL DESIGN

4.1 General Test Design

The Safe Building Monitoring and Detection Technology Verification Program of EPA's NHSRC addresses a relatively broad scope of chemical vapor detection applications. Three main use-concepts can be envisioned: (1) detect-to-warn, (2) detect-to-respond, and (3) detect-to-restore. These different use concepts have different requirements, and thus, permit potentially different technologies (or configurations of a single detection technology) to be considered for each application. For example, detect-to-warn would require permanently installed, continuously operating systems that are integrated into the building's infrastructure and utilities. Instruments used by a first responder, however, need to be fast-responding and portable (i.e., light in weight, battery-powered) and are used on demand. Instruments used in restoration (i.e., post-decontamination) need be neither fast nor portable, but would need to have low detection limits to determine whether an area is clean. Similarly, the range of environmental operating conditions can be different in these different use scenarios.

The use scenario of detect-to-respond was chosen as the focus of this test/QA plan for portable IMS technologies. The performance parameters to be verified and the test conditions are therefore intended to be relevant to use by a first responder, or other personnel needing rapid, real-time indication of an immediate hazard.

The general test design is to first benchmark instrument performance when operated according to the manufacturers' instructions. This will include following manufacturers' recommendations for calibration, warm-up time, and operating conditions (e.g., ambient temperature range). The challenge vapor concentration most relevant to a first responder is the immediately dangerous to life and health (IDLH) level, and consequently concentrations approaching this level will be used in benchmark experiments with a variety of chemicals and chemical agents. Normal indoor air temperature and RH will be established for these benchmark experiments. In addition to the benchmark experiments to establish response time and characterize IMS instrument performance, test conditions will be varied to explore the IMS

response threshold, and to assess the impact on IMS instrument response of realistic stresses or ranges of conditions likely to be encountered during actual field use. For example, cold-start operation (not allowing proper warm-up time), startup after hot storage, differing temperature and humidity conditions, and the introduction of potentially interfering compounds, are all included in the test matrix.

A description of the performance parameters to be characterized and the rationale for their inclusion is provided in Sections 4.2 and 4.3. The chemicals of interest that will be used for the vapor challenges are discussed in Section 4.4. The test matrix and schedule are discussed in Sections 4.5 and 4.6, respectively, and the reference methods to be used are introduced in Section 4.7. Greater detail on the test procedures is given in Section 6 of this test/QA plan.

4.2 Performance Parameters

The key performance parameters to be evaluated in this verification test are:

- Response time
- Recovery time
- Accuracy
- Repeatability
- Response threshold
- Temperature and humidity effects
- Interference effects
- Cold/hot start behavior
- Battery life.

Most of these performance parameters will be evaluated with TICs and with CW agents as the target analytes. However, cold/hot start behavior and battery life will be evaluated only with a single TIC as the target analyte. These performance parameters are defined, and general test procedures are outlined, in Sections 4.2.1 to 4.2.9. Specific test procedures to evaluate these parameters are in Sections 6.1 to 6.9. In addition to these key performance parameters, operational characteristics of the units will be recorded. These operational characteristics include:

- Ease of use
- Signal/data output
- Cost.

These characteristics will be evaluated based on operator observations and available information on the IMS instruments.

4.2.1 Response Time

The determination of IMS response time will accommodate the variety of responses and displays provided by commercial IMS instruments. Consistent with the first response scenario, response time will be determined as the time until the instrument produces an alarm indicating the detection of the challenge chemical, after the introduction of a step change in the concentration of the challenge chemical. The response time will be measured from the start of a fixed challenge vapor concentration, after the IMS instrument has been stabilized by sampling a clean air stream. If multiple forms of response (e.g., an alarm and a scale reading) are outputs of the device, then both will be recorded. The final stable reading of the IMS instrument will also be recorded, whether that reading is in the form of a quantitative measurement or a qualitative (e.g., low/medium/high) response.

The response time is to be verified because a rapid indication of chemical concentration will be needed by first responders to assess the potential hazard.

4.2.2 Recovery Time

Recovery time (or clear-down time) is defined as the time for the IMS instrument to return to its baseline reading (established prior to exposure to the challenge vapor), after it has reached stable readings while sampling the challenge vapor. This performance parameter will be verified for devices that provide a quantitative output, as well as for those that only produce a qualitative or semi-quantitative output. Consistent with the first response scenario, recovery time will be determined as the time between removing the challenge vapor concentration and the cessation of the IMS instrument's alarm.

Recovery time is being verified to illustrate how much time the IMS instrument requires to clear before it is ready to provide an accurate reading in another sampling event. This factor would be relevant when a first responder enters an area that causes an alarm. The IMS instrument would have to clear (i.e., stop giving an alarm) before it could be used reliably in another area in the building.

4.2.3 Accuracy

Accuracy is defined as the degree of agreement between the chemical concentration indicated by an IMS instrument and that measured by a reference method. Accuracy will be verified by direct comparison of reference and IMS data only for those IMS instruments that output a quantitative response as an analog or digital signal. For IMS instruments that output only audible or visual alarms, accuracy will be determined relative to the response threshold in terms of correct (or false) positive and negative indications of the presence of the target chemical. IMS instruments that identify the chemical being sensed will also be evaluated relative to accurate identification of the chemical.

The accuracy of IMS instruments that indicate a relative concentration by status bar or low/medium/high indicators will be determined based on the correlation of indicator reading to concentration provided by the vendor, if such correlation information would be available to a user of the instrument in a first response situation. For example, if the transition to a "high"

reading is programmed to occur at concentration X, then the IMS detector will be credited with an accurate reading whenever it reports a "high" response at an analyte concentration equal to or greater than X.

Accuracy is being assessed to demonstrate that the indicated response is a true indication of the actual vapor challenge concentration.

4.2.4 Repeatability

Repeatability is defined as the consistency of the IMS instrument's indicated response to a constant vapor challenge concentration. Repeatability defined in this way applies to IMS instruments that output a concentration reading in the form of an analog or digital signal, status bars, or a qualitative audible or visual alarm.

Repeatability is being assessed to provide the prospective IMS user with information on the consistency of response at constant vapor concentrations.

4.2.5 Response Threshold

The IMS instrument's response threshold is defined as the approximate concentration that causes the instrument to indicate a response above the baseline reading obtained when sampling clean air at the target test conditions. For instruments that provide a continuous quantitative reading, the response threshold will be the minimum concentration that produces readings uniformly above the zero level. For IMS instruments that provide a relative measure of response such as a status bar or "low/medium/high," the response threshold will be defined as that concentration required to indicate the next highest reading above the baseline. The response threshold for IMS instruments that provide an audible or visual alarm will be that minimum concentration required to cause the audible or visual alarm.

The response threshold is being assessed to determine whether the IMS instruments have adequate sensitivity to chemicals of interest. A precise determination of the response threshold is not needed because the first responder will be using the IMS instrument to determine an

immediate hazard, rather than an exact concentration. Therefore testing that brackets the response threshold within an approximate range is considered sufficient.

4.2.6 Temperature and Humidity Effects

The effect that the temperature and RH of the sampled atmosphere have on IMS instrument response will be evaluated. In all cases, the IMS instrument undergoing testing will be maintained at the same temperature as the challenge air stream. The challenge air stream also will be maintained at the specified RH.

The temperature and RH conditions to be used in testing were selected based on those likely to be experienced in an indoor environment in actual use by first responders. In the event of a chemical release it is possible that the windows of a building could be opened to flush out the contaminant. Conversely, safe building protocols also may require closing a building to prevent infiltration of outside vapor hazards, to minimize exposure of the surrounding populace, or to minimize convective transport of contaminants throughout a building. Overall, it is unlikely that the indoor building conditions encountered by a first responder would range over the full extremes of potential outdoor conditions. Consequently, a narrower range of temperature and RH is considered appropriate for this verification test, as indicated in Table 2. Each "X" in a cell in Table 2 indicates a condition of temperature and RH that will be used as a test condition in this verification test.

Temperature and RH effects are being assessed to establish whether IMS readings are influenced by environmental conditions during use.

Table 2. Temperature and Relative Humidity Conditions for Portable IMS Instrument Testing

	Temperature (°C)		
RH (%)	**5 ± 3**	**22 ± 3**	**35 ± 3**
≤ 20		X	
50 ± 5	X	X	X
80 ± 5		X	X

4.2.7 Interference Effects

The effect of potentially interfering compounds present in the indoor building atmosphere will be assessed. The selected potential interferents are a diverse set of chemicals that could be ubiquitous in buildings under a first-response scenario, and whose presence is not seasonally dependent. The representative set of potentially interfering compounds to be used in testing are as follows: (1) ammonia-based cleaner, (2) latex paint fumes, (3) gasoline vehicle exhaust, (4) air freshener vapors, and (5) N, N-diethylaminoethanol (DEAE), a common additive in building boiler systems that can be a ubiquitous indoor contaminant. These potential interferents will be tested both with and without the target TICs and CW agents present, at the normal temperature and RH conditions (22ºC and 50% RH).

The effect of potentially interfering compounds is being assessed because such compounds can potentially produce two types of errors with IMS instruments: (1) erroneous reporting of the presence of a chemical or chemical agent when none is present (false positives) or (2) reduction in sensitivity or masking of target analytes of interest (false negatives). False positives will be assessed by alternately sampling clean air and air containing the interferent, in the absence of any target chemical or agent. False negatives will be assessed by alternate sampling of clean air and air containing both the interferent and a target chemical or agent. Both types of tests will be conducted with each of the interferent species and each of the target chemicals and agents.

4.2.8 Cold/Hot Start Behavior

The test of cold start behavior will assess how the IMS response to a target challenge concentration at baseline environmental conditions is affected when the IMS instrument is not permitted adequate warm-up time per the manufacturer's instructions. The performance of the IMS devices will be evaluated without any warm up period, to simulate the effect of immediate use that could be required in an emergency. The time delay between turning the IMS instrument on and when the IMS instrument is ready to begin giving any reading at all will be a primary

factor determined in this test. In addition, as appropriate for the IMS instrument being tested, the response time to a vapor challenge, and the accuracy of readings relative to the challenge concentration will be evaluated. The cold start behavior will be evaluated with both a cold start from room temperature and from reduced temperature (i.e., after storage of the IMS instruments overnight in a refrigerated environment at 5 to 8°C). Conversely, a hot soak followed by startup is also of interest, because IMS instruments may be stored/transported in vehicles parked in the sun. Such heat exposure may affect performance, especially electronics. Note that the "hot start" evaluation means that the IMS instrument is taken from storage in a hot environment and then started; it is not "hot" in the sense of having been running previously. The hot soak will consist of storing the IMS instruments overnight at a temperature of 40 ± 3°C before testing. As in the cold soak tests, the response time and accuracy of readings will be assessed. A single cold start test will be conducted from each of these three aforementioned starting conditions (room temperature, 5-8ºC, and 40ºC), using hydrogen cyanide (AC) as the challenge TIC.

Vendors have indicated that actual use conditions and operating parameters are not and cannot always be followed by the emergency responders. Therefore, IMS instruments may be used in a fashion that is not ideal. The need for immediate readings upon arrival at an emergency is the motivation for testing cold/hot start behavior.

4.2.9 Battery Life

Portable IMS instruments will typically be battery operated and thus performance will be dependent on proper performance of the batteries. Battery life is defined as the amount of time the IMS instrument can operate on fully charged or new batteries. A one-time test will be conducted to determine how long the instrument will run on a single, full charge or one set of new, disposable batteries. This test will involve repeatedly monitoring a challenge level of hydrogen cyanide (AC) until the instrument's batteries are depleted.

4.3 Operational Characteristics

Key operational characteristics of the IMS instruments will be evaluated by means of the observations of test operators, and by inquiry to the IMS vendors.

Ease of use will be assessed by operator observations, with particular attention to the conditions of use by first responders. For example, the use of PPE (e.g., gloves, respirator) may make it difficult to turn the instrument on or off, operate it, or read the display. These factors will be assessed by outfitting an operator with such PPE, and noting any difficulties in operating the instrument. This assessment will be done separately from any test of the other performance parameters with TICs or CW agents.

Signal or data output capabilities of the IMS instruments will be assessed by observations of the testing personnel who operate the instruments during testing. The type of data output will be noted (e.g., audio or visual alarm, bar graph, low/med/high indication, quantitative measure of concentration, etc.). In addition, the clarity and readability of the output will be noted, especially in low light conditions or when holding the IMS instrument while walking, as in use by a first responder. The availability of multiple forms of data output or display also will be assessed, e.g., the availability of both a visual display and an analog voltage output for recording purposes.

Costs for each IMS instrument will be assessed by asking the vendor for the purchase and operational costs of the instrument as tested in this program. This verification test will not be of sufficient duration to test long-term maintenance or operational costs of the IMS instruments. Estimates for key maintenance items will be requested from the vendors to address those costs. Costs will be those at the time the IMS instruments are made available for testing.

4.4 Chemical Test Compounds

This test/QA plan cannot consider all the chemicals that a first responder could potentially encounter when responding to a possible vapor hazard in a building. An emergency response may be necessary due to an accidental spill of relatively innocuous chemical, or to a purposeful release of a hazardous chemical. One focus of chemical selection in this plan is on a

set of TICs commonly considered by the DoD community as potential hazards. Initial experiments will challenge the IMS instruments with selected TICs. After completing TIC experiments, the IMS instruments will be challenged with CW agents. The TICs selected for use in this verification are (chemical formula and agent designation in parenthesis): cyanogen chloride (ClCN; CK), hydrogen cyanide (HCN; AC), phosgene ($COCl_2$; CG), chlorine ($C1_2$; no military designation), and arsine (AsH_3; SA). The CW agents selected for use in testing are sarin (GB) and sulfur mustard (HD).

4.5 Test Matrix

Table 3 summarizes the evaluations to be conducted in this verification test. As Table 3 indicates, except for cold start and hot start behavior, battery life, and assessment of false positive interference effects (i.e., the interferent alone), all performance parameters will be evaluated with all five TICs and with both CW agents. Cold and hot start behavior and battery life will be tested only with hydrogen cyanide (AC) as the target TIC.

4.6 Test Schedule

Testing under this test/QA plan is expected to begin in July, 2004. It is anticipated that about two months will be required to complete all TIC testing for a single IMS technology. This schedule is predicated on the first IMS vendor providing two of their IMS instruments for testing by early June 2004. Because effort and resources are required to construct test fixtures for controlled challenge atmosphere generation, a test apparatus will be constructed for testing one chemical at a time. Testing will then consist of sequencing through the TICs at Battelle's King Avenue laboratories, and testing with the CW agents at the Battelle chemical surety laboratory. Testing the TICs first allows for the most rapid and cost effective means to conduct tests. If any equipment (reference instrument or test fixture) maintenance or modification is required, it will be easiest to do it prior to CW agent exposure. Testing with TICs will initially emphasize the

Table 3. Summary of Evaluations to be Conducted in Portable IMS Detector Verification Test

Performance Parameter	Objective	Comparison Based On
Response Time	Determine rise time of IMS response	IMS readings with step rise in analyte concentration
Recovery Time	Determine fall time of IMS response	IMS readings with step decrease in analyte concentration
Accuracy	Characterize agreement of IMS with reference results	Reference method results
Repeatability	Characterize consistency of IMS readings with constant analyte concentration	IMS readings with constant input
Response Threshold	Estimate minimum concentration that produces IMS response	Reference method results
T and RH Effects	Evaluate effect of T and RH on IMS performance	Repeat above evaluations with different T and RH
Interferent Effects	Evaluate effect of building contaminants that may interfere with IMS performance	Sample interferents and TICs/CW agents together (and interferents alone[a])
Cold Start	Characterize startup performance of IMS instruments	Repeat tests with no warmup[a]
Hot Start	Characterize startup performance after hot storage	Repeat tests with no warmup[a]
Battery Operation	Characterize battery life and performance	Compare results on battery *vs* AC power[a]

a: Indicates this part of the test not performed with CW agents.

baseline environmental conditions of 22 ± 3°C and 50 ± 5% RH. The procedures for temperature and RH effects and the interferent tests will be conducted following the initial benchmark experiments.

Figures 2 and 3 illustrate the logical stepwise progression of test procedures in this verification. These figures show that most procedures are conducted both with TICs and with CW agents. However, the cold and hot start tests, and battery life test are conducted only with a single TIC.

Sections 6.1 through 6.9 of this plan describe how each of the procedures in Figures 2 and 3 will be performed.

Test 1: Vapor Challenge with TIC Alternating clean air with IDLH level concentration of TIC five times with IMS detector operating fully warmed up per manufacturer's instructions prior to testing, and room temperature (22 ± 3 °C) and 50 ±5 %RH.
Test 2: Vapor Challenge with TIC at reduced concentration Test 1 is repeated at a lower concentration giving mid-range on-scale readings (only if off-scale response at IDLH).
Test 3: Vapor Challenge with TIC at increased concentration Test 1 is repeated at roughly 10 times the IDLH concentration (only if no response at IDLH).
Test 4: Response Threshold of TIC Test 1 is repeated at a concentration below IDLH. If a response is recorded, the concentration is cut in half until no response is recorded. If no initial response is recorded, the concentration is increased by a factor of 2 until a response is recorded.
Test 5: IDLH/0.1 IDLH/Clean Air Challenge Test 1 is repeated by cycling among IDLH, a low concentration (either 0.1 IDLH or response threshold), and clean air six times, and alternating order of IDLH and low concentration.
Test 6: Vapor Challenge with TIC at room temperature, low humidity Test 1 is repeated at room temperature (22 ± 3 °C) and less than 20% RH. The test is performed at the concentration(s) determined via the logic in Figure 3.
Test 7: Vapor Challenge with TIC at room temperature, high humidity Test 1 is repeated at room temperature (22 ± 3 °C) and 80 % RH. The test is performed at the concentration(s) determined via the logic in Figure 3.
Test 8: Vapor Challenge with TIC at high temperature, medium humidity Test 1 is repeated at high temperature (35 ± 3 °C) and 50 % RH. The test is performed at the concentration(s) determined via the logic in Figure 3.
Test 9: Vapor Challenge with TIC at high temperature, high humidity Test 1 is repeated at high temperature (35 ± 3 °C) and 80 % RH. The test is performed at the concentration(s) determined via the logic in Figure 3.
Test 10: Vapor Challenge with TIC at low temperature, medium humidity Test 1 is repeated at low temperature (5 ± 3 °C) and 50 % RH. The test is performed at the concentration(s) determined via the logic in Figure 3.
Test 11: Interferent false positive tests Test 1 is repeated alternating interferent only with clean air. The test is repeated for all interferents in both libraries.
Test 12: Interferent false negative tests Test 1 is repeated alternating TIC and interferent with clean air. The test is repeated for all interferents.
Test 13: Opposite Library test Test 1 is repeated for the library opposite of the one recommended by the manufacturer for TICs.
Test 14: Room Temperature, cold start behavior Repeat Test 1 with the IMS detector at room temperature for a minimum of 12 hours and no warm-up.
Test 15: Cold-/Cold-start behavior Repeat Test 1 after the IMS detector has been kept refrigerated (5-8 °C) for a minimum of 12 hours, with no warm-up.
Test 16: Hot-/Cold-start behavior Repeat Test 1 after the IMS detector has been kept heated (40 °C) overnight for a minimum of 12 hours, with no cool down or warm up.
Test 17: Battery test Repeat Test 1 with the IMS detector operating on battery power. The TIC at IDLH concentration is alternated with clean air once every half hour until the unit stops responding or shuts down due to loss of power.

Figure 2. Test Sequence for IMS Instrument Verification

Step 1: Perform Test 1. Depending on the results of this test, go to Step 2a, 2b, or 2c as appropriate.
Step 2a: If there is no response in Test 1, perform Test 3, then go to Step 4.
Step 2b: If the response in Test 1 is on scale, then skip to Step 3 and perform all subsequent tests at the IDLH concentration.
Step 2c: If the response in Test 1 is full- or off-scale, perform Test 2. Establish the concentration that gives a mid-range on-scale response, and then proceed with Step 3, using that established concentration in all subsequent tests.
Step 3: Perform Test 4 (if not already done), Tests 5-10, Tests 12-13 at the concentration(s) determined above. For the first TIC, also perform Test 11 and Tests 14-17.
Step 4: Return to Step 1 and repeat Tests 1 through 13 for all other TICs.
Step 5: Repeat Tests 1 through 13 for all CW agents

Figure 3. Stepwise Logic for IMS Instrument Verification

4.7 Reference Methods

Table 4 summarizes the reference methods to be used for determining the challenge concentrations of the target TICs and CW agents in the test. Listed in the table are the target TICs and CW agents, the sampling and analysis methods to be used for each compound, and the applicable concentration range of each method. References to the method used are footnoted in Table 4. For the TICs cyanogen chloride and hydrogen cyanide, samples will be injected directly for determination by gas chromatography (GC) with flame ionization detection (FID).[2,3] Phosgene will be determined by a colorimetric method using a liquid reagent solution in a small impinger train.[4] Chlorine will be determined by a commercially available continuous electrochemical analyzer with a chlorine-specific sensor (Draeger Mini Warn, or similar), to allow rapid determination of chlorine levels delivered to the IMS instruments during testing. Arsine will be determined by a gas chromatographic method with a capillary column and mass selective detection (MSD), using samples collected by syringe from the test apparatus.[5] A retention time of about seven minutes is expected for arsine, allowing repeated analysis within each test procedure.

The CW agents GB and HD will be collected on solid sorbent cartridges, and determined by GC with flame photometric detection (FPD). Determination of the CW agents will be conducted according to the procedures and quality requirements of *HMRC Standard Operating Procedure (SOP) "HMRC-IV-056-07,* for *Operation and Maintenance of Gas Chromatographs and for the Analysis of Solutions Containing GA, GB, GD, GF, HD, and VX by Gas Chromatography."* The procedures of this method for gas chromatographic (GC) analysis will also be adapted for the analysis of TICs by GC.

The concentrations of most interferents used in the verification will be checked by means of a continuous total hydrocarbon (THC) analyzer. The target concentrations to be used are indicated in Section 6.7. The interferent DEAE is the exception to this procedure. Its target concentration is too low to be monitored with the THC analyzer, however the DEAE concentration will be established based on dilution of a known DEAE standard mixture.

Table 4. Planned Reference Methods for Target TICs and CW Agents

Analyte	Concentration Range (ppm)	Sampling Method	Analysis Method
Cyanogen chloride (CK)	2 ~ 100	Air sample injected directly	GC/FID[a]
Hydrogen cyanide (AC)	0.05 ~ 100	Air sample injected directly	GC/FID[b]
Phosgene (CG)	1 ~ 100	Collection in impingers with nitrobenzyl pyridine reagent	Visible absorption at 475 nm[c]
Chlorine (Cl_2)	0.1 ~ 100	Continuous electrochemical detector with chlorine-specific sensor	Continuous detection[d]
Arsine (SA)	0.05 ~ 100	Capillary gas chromatography with direct injection	Mass selective detector (MSD)[e]
Sarin (GB)	0.01 ~ 100	Air sample collected on solid sorbent	Thermal desorption, GC/FPD[f]
Sulfur mustard (HD)	0.01 ~ 100	Air sample collected on solid sorbent	Thermal desorption, GC/FPD[f]

a: See reference 2.
b: See reference 3.
c: See reference 4.
d: Commercially available detector.
e: See reference 5.
f: These measurements governed by HMRC SOP HMRC-IV-056-07.

5.0 MATERIALS AND EQUIPMENT

5.1 Agents and TICs

As noted in Section 4.4, the chemical TICs to be used in this verification test will include: cyanogen chloride (CK), hydrogen cyanide (AC), phosgene (CG), chlorine, and arsine (SA). These gases are relatively common and readily available materials that could be used by terrorists to attack a building. Chlorine is also a common, high-volume industrial chemical that might be found at the scene of an industrial accident or transportation spill. All TICs except cyanogen chloride will be purchased as dilute compressed gas mixtures from commercial vendors, with a balance of nitrogen. The concentrations of those mixtures will be determined based on the required challenge target concentrations. For cyanogen chloride, a compressed gas standard will be prepared in Battelle's laboratories, using neat cyanogen chloride as the starting material.

The CW agents planned for use in the verification test include sarin (GB) and sulfur mustard (HD). These agents are reasonable potential threats, and have been used in previous tests of CW agent detectors for military applications, thereby providing a possible link between this verification test and previous testing. The CW agents will be obtained from the U.S. Army, under the bailment agreement noted in Section 3.2.

5.2 Vapor Delivery Equipment

Different vapor delivery equipment will be used depending on the TIC or CW agents to be tested. Compressed gas cylinders will be used as the vapor delivery source for all the TICs: cyanogen chloride, hydrogen cyanide, phosgene, chlorine, and arsine. For the less volatile CW agents GB and HD, a diffusion cell will be used. A temperature controlled water bath will be installed to control the temperature of the diffusion cell, to maintain a stable and controllable vapor generation rate. Suitable valving will be included in the flow path downstream of the vapor generation source, so that the dilution and test equipment can be totally isolated from the

source if necessary. A schematic of the entire vapor generation, dilution and delivery system is shown in Section 6.0.

5.3 Temperature/Humidity Control

The IMS instruments will be evaluated at temperatures specified in Table 2, Section 4.2.6. Both the delivered air temperature and the IMS instruments will be maintained within the specified temperature range. For testing at 35°C, the vapor delivery system will be warmed with heat-traced line, using an electronic temperature controller. For testing at 5°C, the dilution and delivery system will be enclosed in a cooled chamber, to provide approximate temperature control. For all tests, thermocouples will be installed in both the clean air plenum (see Section 6.0) and the challenge plenum to provide real-time temperature monitoring.

A commercial Nafion® humidifier (Perma Pure, Inc.) will be used to generate controlled high humidity air (50 to 100% RH), which will then be mixed with dry dilution air and the target vapor stream to obtain the target RH (≤ 20% to 80%) in the challenge air.

5.4 Reference Methods

The planned reference methods were summarized in Section 4.7. The media used will depend on the analyte and concentration range of interest. In summary, gas samples for CK and AC will be determined by direct injection via sample loop with analysis by GC/FID. Phosgene will be determined by impinger collection and measurement of visible absorption at 475 nm. For arsine, direct injection via syringe will be used, for analysis by GC/MSD. Chlorine will be determined continuously by a chlorine-specific electrochemical sensor. For the CW agents, samples will be collected onto commercially available solid sorbent cartridges, and subsequently thermally desorbed and injected for GC/FPD analysis.

5.5 Performance Evaluation Audit

The equipment needed for conducting the performance evaluation audit will consist of independent standards used to check the reference methods against which IMS detector responses are compared. The PE audit will be conducted only for four of the five TICs. No PE audit will be conducted for cyanogen chloride (CK), since the standard for this TIC is made by Battelle from pure starting materials, and no independent standard is available. For the other TICs (AC, CG, SA, and Cl_2), the independent standards will be gaseous standards of the target TICs, obtained from different commercial suppliers than those providing the standards used for reference method calibrations. Also, no independent PE standards are available for the CW agents, i.e., all CW agents used in testing are obtained from the U.S. Army. In lieu of a true PE audit, one or more QA check samples will be prepared for each CW agent used in testing, by spiking sorbent traps and analyzing them by the same method used to analyze test samples. Description of the criteria for the PE audit is provided in Section 7.2.2.

6.0 TEST PROCEDURES

The schematic of the test system is illustrated in Figure 4. The test system consists of a vapor generation system, a Nafion® humidifier, two challenge plenums, a clean air plenum, RH sensors, thermocouples, and mass flow meters. The challenge vapor or gas is generated by the vapor generation system. The appropriate vapor generator, such as a diffusion cell or compressed gas cylinder, will be selected depending on the compound of interest and the concentration range to be tested. The challenge vapor from the vapor generation system will then mix with the humid dilution air and flow into the challenge plenum.

As illustrated in Figure 4, the RH of the challenge mixture will be set by adjusting the mixing ratio of the humid air (from the Nafion® humidifier) to the dry dilution air, and the concentration of the challenge gas or vapor will be set by adjusting the ratio of the gas or vapor generation stream to the humid dilution air stream, respectively. To avoid potential corrosion or malfunction of the relative humidity sensor from exposure to the challenge gas or vapor, the RH meter will be installed upstream of the inlet of the challenge vapor stream. The RH of the final challenge stream will be calculated based on the measured RH of the humid dilution air, and the mixing ratio of the vapor generation stream to the humid dilution air.

To establish the background readings of the two IMS units being tested, a clean air plenum will be installed. Part of the humid dilution air will be introduced directly into the clean air plenum. When establishing the IMS instrument background, the four-way valves connected to the two IMS units will be switched to the clean air plenum to collect baseline data.

After the background measurement, the four-way valves connected to the two IMS units will be switched to the challenge plenum to allow the IMS instruments to sample the challenge mixture. Switching between the challenge and clean air plenums will be rapid, and the residence time of gas in the test system will be short, to allow determination of the response and recovery times of the IMS instruments. The use of two challenge plenums allows an assessment of the recovery of IMS response, as when the user moves from one contaminated area to another area

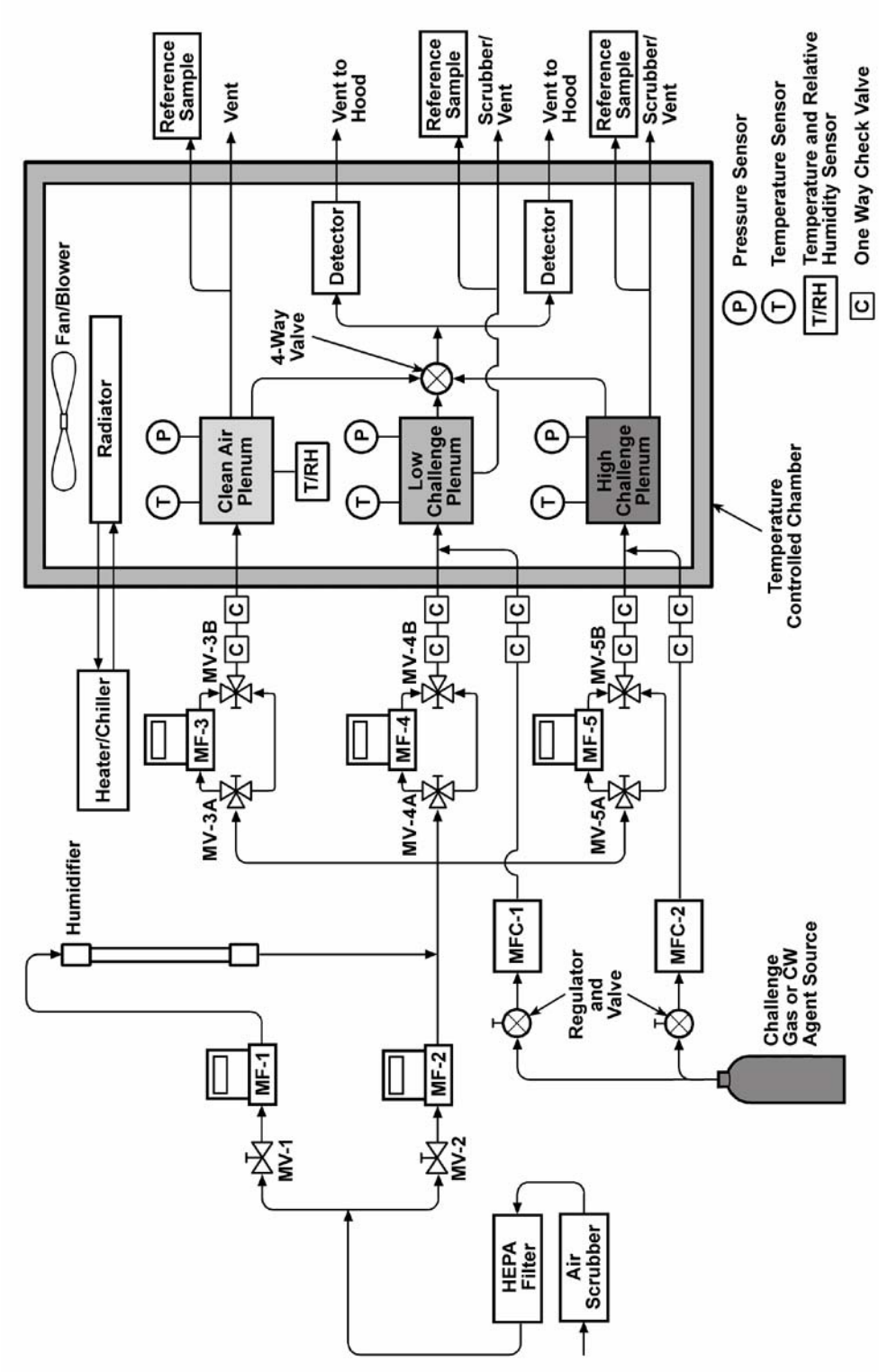

Figure 4. Test System Schematic

of different contaminant concentration. The reference methods described in Section 4.7 will be used to quantify the gas or vapor concentrations in the clean air plenum and the challenge plenums to provide a cross-check of the concentrations measured.

The test system depicted in Figure 4 is the basic system that will be used to assess the response and performance of IMS instruments to challenge mixtures of the selected chemicals. The specific components and methods will depend, in part, on the type of evaluation and chemical challenge. For example, the system will draw a known flow of the target chemical from a compressed gas cylinder, when testing with a volatile chemical such as the TICs, or use a diffusion cell for less volatile compounds such as the CW agents. Similarly, the test system will also incorporate an interferent generator (not shown in Figure 4) as needed in the test for evaluation of interference effects. The interferent generator will be a simple but realistic vapor source, for delivery of paint fumes, ammonia cleaner vapors, and air freshener vapors. For these interferents, a flow of approximately 100 cm^3/min of clean air will be passed through a sealed glass vessel containing a stirred aliquot of the interferent material. The vapor picked up by the air stream will be diluted in the air flow to the test plenum, to achieve the target interferent concentrations specified in section 6.7. For delivery of vehicle exhaust, the interferent source may be a small flow of whole exhaust or a compressed gas mixture containing key chemical components of the exhaust. Testing with DEAE will use a compressed gas mixture of DEAE in nitrogen, prepared in Battelle's laboratories. The same interferent sources will be used in all tests.

The test system will be constructed so that a dedicated clean air stream and one or more challenge air streams can be sampled. The dedicated streams are needed to properly establish the system response to clean air prior to an experiment. This is critical when testing a parameter such as response time, so that the time constant of the test system can be uncoupled from that of the instrument. A single stream system would require too much time to change from clean air to challenge air, preventing the actual response time of the IMS instrument from being properly measured. Furthermore, the means of supplying the challenge air streams to the IMS instrument must provide those sample streams at ambient atmospheric pressure, i.e., without increasing or decreasing the pressure of the IMS inlet. The exact means of connecting an IMS instrument to

the test apparatus in Figure 4 will vary depending on the instrument's inlet design, but that connection must prevent over- or under-pressurization.

6.1 Response Time

To evaluate IMS response time, the environmental conditions will be established at the target conditions of 22 ± 3ºC and 50 ± 5% RH. Initially 10 L/min of the clean humidified air will pass through the clean air plenum. The IMS instruments will sample the clean air for a minimum of 30 seconds, or until a stable reading has been indicated, but not to exceed 10 min, to obtain a baseline for the IMS instrument. A stable reading is defined as one that does not change when all system conditions are unchanged. For IMS instruments that do not provide an analog or digital signal, but rather a status indicator such as a meter bar or relative measure (e.g., low/medium/high), IMS readings will be considered stable when there is no change in the reading over a 1-minute period. If the IMS instrument has a digital or analog signal, readings that fluctuate by less than ± 20% and show no apparent trend over a 1-minute period will be considered stable. The clean air plenum will also be sampled with the appropriate reference method at least once during this test procedure. This sampling will take place after IMS readings have been stabilized.

Concurrent with the background measurements will be the establishment and demonstration of the target challenge concentration in the high challenge plenum. The high challenge concentration will be generated at the target environmental conditions. For the TICs, adjustments will be made to the generator operating conditions and the dilution flow as needed to establish a challenge concentration within ± 20% of the IDLH or other target, with a stability characterized by a percent relative standard deviation of 10% or less in successive reference measurements. For the CW agents, a delivered concentration within 35% of the target level will be deemed acceptable. Reference samples will be collected and analyzed immediately to establish the challenge concentration and demonstrate stability. Testing may commence before reference analyses are completed, provided the staff conducting testing have substantial confidence based on other measurements (e.g., gas flow rates) that the challenge concentrations

are within the target specifications. However, if the reference analyses upon completion show the challenge concentration to be outside the target specifications, then the affected portion of the test procedure must be repeated with the correct challenge concentration. A challenge concentration will be considered stable if it can be maintained within the target challenge bounds based on two consecutive reference sample measurements prior to the test.

After a stable reading is obtained from the IMS instruments on background air, and the challenge mixture is stable and at the target concentration, the four-way valve at the IMS instrument's inlet will be switched to sample from the challenge plenum. The response of the IMS instruments will be recorded and the time to produce an alarm from the instrument will be determined as the response time. When feasible based on the time response of the reference method, the challenge vapor concentration will also be determined by reference method sampling periodically during the procedure. The IMS will sample from the challenge plenum for a minimum of 30 seconds, or until a stable reading is obtained, up to a maximum of 10 min.

After the challenge sampling has concluded, the sample inlet four-way valve will be switched to again sample from the clean air plenum. The time required for the IMS instruments to clear, i.e., the time to return to starting baseline or non-alarm readings, will be recorded as the recovery time. After a maximum of 10 min, regardless of whether the IMS instrument has returned to baseline, subsequent cycles of alternating challenge/clean air sampling will be carried out, controlled by the 4-way valve. A total of five such challenge/clean air cycles will be completed.

In the case of an instrument that enters a "backflush" mode or otherwise interrupts sampling upon detection of the target chemical, a different approach will be used from that outlined above. Upon interruption of sampling due to detection of the chemical, the instrument will immediately be switched back to sampling from the clean air plenum. That is, the requirement for a minimum 30 second sampling period will be removed. Once the interruption or "backflush" has ended, the baseline measurement will be taken and the process repeated.

Following the five challenge/clean air cycles, a set of six cycles will be conducted in which the IMS instruments sample sequentially from the high, low, and clean air challenge plenums. The high challenge plenum will provide the IDLH or other primary target

concentration, and the low challenge plenum a concentration of approximately 0.1 times that level, or the response threshold (see Section 6.5), whichever is greater. Clean air will be sampled alternately with sampling from the challenge plenums, and the order of sampling from the high (H) and low (L) challenge plenums will be reversed, i.e., the order of sampling will be clean air/H/L/clean air/L/H/clean air/H/L/... for a total of six such cycles. This procedure will simulate use of the IMS instruments in locations having different degrees of contamination. If necessary, the alternate procedure described above for instruments that interrupt sampling or go into a "backflush" mode will be used in this test as well.

The same sampling procedure will be carried out at different temperature and RH conditions or challenge concentration to evaluate temperature and RH effects and response thresholds. The initial test will be conducted at a concentration equal to the target chemical's IDLH level. If the chemical does not have an IDLH, then another concentration of significant health impact will be targeted. These levels are shown in Table 5. The temperature and humidity effects will similarly be assessed using the IDLH or other significant concentration.

If the instrument gives a full scale or overscale reading when challenged at the IDLH level at the normal temperature and RH conditions (22ºC and 50% RH), then a lower challenge concentration will be chosen that provides an on-scale reading. All subsequent tests for that TIC or CW agent will then use that lower challenge concentration. If the instrument does not respond to the IDLH or other initial concentration selected, then all subsequent tests planned for that TIC or CW agent will be eliminated. Otherwise, testing will proceed as described.

Table 5. Target Challenge Concentrations

Chemical	Concentration	Type of Level
Cyanogen chloride (CK)	20 ppm (50 mg/m^3)	Estimated based on IDLH for HCN
Hydrogen cyanide (AC)	50 ppm (50 mg/m^3)	IDLH[a]
Phosgene (CG)	2 ppm (8 mg/m^3)	IDLH
Chlorine (Cl_2)	10 ppm (30 mg/m^3)	IDLH
Arsine (SA)	3 ppm (10 mg/m^3)	IDLH
Sarin (GB)	0.035 ppm (0.20 mg/m^3)	IDLH
Sulfur mustard (HD)	0.09 ppm (0.6 mg/m^3)	AEGL-2[b]

a: IDLH = Immediately dangerous to life and health
b: AEGL = Acute Exposure Guideline Level; AEGL-2 levels are those expected to produce a serious hindrance efforts to escape in the general population.[6] The values shown assume a 10-minute exposure.

6.2 Recovery Time

The time for the IMS instrument to return to its baseline reading or non-alarm state after removing a challenge concentration will be measured as described under Response Time, Section 6.1. No additional tests are planned beyond those conducted in Section 6.1.

6.3 Accuracy

The accuracy of the IMS instruments will likewise not require any additional tests. In all the response threshold and response time tests, the challenge concentration will be measured using a reference method or monitor. Reference samples will be collected along with all IMS testing to ensure that a stable concentration is maintained. The reference samples will be the ground truth samples used to assess accuracy for those IMS instruments that give a quantitative concentration reading. For IMS instruments that give only a relative indication of concentration, such as indicator bars, accuracy will be assessed based on manufacturer-supplied data on the relationship of instrument readings to analyte concentration, if possible. It is assumed that manufacturers have correlated such readings to absolute concentrations during development. If those data are not proprietary and are provided, they will be used to assess accuracy. Alarm readings, initiation of backflush mode, and other IMS responses will be used to assess accuracy as described in Section 8.3.2.3.

6.4 Repeatability

Repeatability will be assessed using data obtained from the repeated clean air/challenge or high/low challenge cycles, in the various tests conducted, such as the response time tests. The repeated test results at the same environmental and concentration conditions will be reported, to demonstrate the repeatability of the measurements. No additional tests specific to this parameter will be performed.

6.5 Response Threshold

The response threshold of each IMS instrument will be evaluated by repeating the procedure of Section 6.1 at successively lower (or, if necessary, higher) concentrations, to define the instrument's response threshold. The response threshold will be determined at the baseline environmental condition of 22 ± 3 °C and 50 ± 5 % RH, in the absence of any interfering chemicals. The manufacturer's reported detection limit (± 50%) will be used as the starting concentration. If no detection limit is reported by the manufacturer, then a concentration at least 10 times lower than the IDLH or other target concentration will be used as a starting concentration. If there is no response at the starting test concentration, then the concentration of the challenge will be increased by a factor of two. Similarly, if the IMS instrument responds to the starting concentration, then the challenge concentration will be decreased by a factor of two. The increase or decrease in concentration will be continued accordingly, until the response threshold has been bracketed. The minimum concentration producing an IMS response will be denoted as the response threshold.

The duplicate IMS instruments tested simultaneously may produce different instrument responses. In that case, the concentrations will be varied as needed to assess the response thresholds of the individual IMS instruments.

6.6 Temperature and Humidity Effects

The tests described under Response Time in Section 6.1 will be repeated at the IDLH or other selected target concentration of significant health concern, over the range of environmental conditions shown in Table 2 (Section 4.2.6). Five repeat runs will be performed at each set of test conditions, for each target TIC or CW agent. The same procedure used in Section 6.1 will be used. The data at different temperature and RH conditions will be used to infer whether these conditions affect the detection (i.e., accuracy, repeatability, response threshold) of the IMS instrument for the target chemical. The effect on response time and recovery time will also be assessed.

6.7 Interference Effects

To evaluate interference effects, the test system shown in Figure 4 will be modified with the addition of an interferent vapor generator. The output from this source will be directed as needed to mix with the humidified air flowing to the challenge plenum. The test chemical generation can be independently controlled such that the interferent will be generated in the absence or presence of the test chemical. This will allow interference effects to be evaluated with the interferent alone, and with each interferent and TIC or CW agent together. Testing with the interferent alone will allow evaluation of false positive responses, and testing with the interferent and chemical together will allow evaluation of false negatives. The test procedures will also allow observation of interferent effects on the response time and recovery time of the IMS instruments. Table 6 shows the target concentrations of the planned interferents. Those concentrations are shown in terms of the equivalent total hydrocarbon concentration in parts per million carbon (ppmC). These target concentrations are based on actual indoor measurements by Battelle, or on published data.

Table 6. Target Concentrations for the Interferents

Interferent	Concentration (ppmC)
Latex Paint Fumes	5-10
Floor Cleaner Vapors	10
Air Freshener Vapors	1
Engine Exhaust	2.5
DEAE	0.02

Interferent testing will involve only one interferent at a time. Testing will be done by alternately sampling clean air and the interferent mixture, for a total of up to five times each, in a procedure analogous to that described in Section 6.1. However, if no interferent effect is observed after three such test cycles, the test will be truncated at that point. Testing with interferents alone will involve alternately sampling from the clean air plenum, and then from the challenge plenum, to which only the interferent in clean air is delivered. The same process will be used for testing with interferents and TICs or agents together, with the two compounds diluted

together in humidified air delivered to the challenge plenum. The same TIC and CW agent concentrations used in the initial testing under Section 6.1 will be used in this test, i.e., the IDLH level or other target level. A response from the IMS instrument with the interferent alone will be recorded as a false positive, and the absence of a response, or a reduced response, to the TIC or CW agent in the presence of the interferent will be recorded as a false negative.

The replicate test runs conducted with the interferent plus TIC or agent will also allow the response time and recovery time of the IMS instruments to be assessed with interferents present. Differences in response and recovery times, relative to those in previous tests with only the TIC or agent present, will be attributed to the effect of the interferent vapor.

6.8 Cold/Hot Start Behavior

The cold/hot start tests will be conducted in a manner similar to the Response Time test in Section 6.1. Prior to these tests, however, the IMS instruments will not be allowed to warm up per the manufacturer's recommendation.

The cold start test will be conducted both with the IMS detectors at room temperature, and subsequently at reduced temperature, prior to startup. In the former test, the IMS instruments will be stored with the power off at 22 ± 3°C for at least 12 hours prior to testing. The cold start effect will be assessed using an IDLH or similar challenge concentration of AC only, at the baseline conditions of 22 ± 3°C and 50 ± 5 % RH. The time from powering up the IMS instruments to their first readiness to provide readings will be determined as the startup delay time. The response time – as defined in Section 6.1 – will be measured, followed by the recovery time. Repeatability and accuracy in five replicate clean air/challenge cycles also will be noted. For the reduced temperature cold start, at the end of the test day the IMS instruments will be placed in a refrigerated enclosure (5 - 8°C) with the power off for at least 12 hours overnight. At the start of the next test day, the cold start test will be repeated, using the same baseline conditions (22ºC and 50% RH) and again recording the startup delay time, and other performance parameters.

For the hot start test, the instruments will be placed in a heated enclosure at 40 ± 3°C with the power off for at least 12 hours overnight. At the start of the next test day, the hot start test will be conducted in the same fashion as the cold start test, at the baseline test conditions (22ºC and 50% RH). Only one cold/hot start test will be performed per day, so that the IMS instruments can equilibrate to storage conditions prior to the test.

The cold/hot start test procedures will be to connect the IMS instruments to the clean air manifold, and switch the instruments on. The time between switching the IMS instruments on and when the instruments indicate they are ready to begin providing readings will be recorded as the delay or standby time for each unit being tested. Then the IMS instruments will be connected (by the four-way valve in Figure 4) to the challenge plenum, which is supplied with the IDLH or other target level of AC. The response time, stable reading, and recovery time of each IMS unit will be recorded, for each of five successive periods of alternating clean air and challenge mixture. The recorded data will be used to evaluate whether response and recovery time, repeatability, and accuracy are affected by a cold or hot start relative to normal (i.e., fully warmed up) operation.

6.9 Battery Life

An evaluation of battery life will be made by assessing the degradation of performance with extended continuous operation. New batteries will be installed, or the IMS instrument batteries will be fully charged. The IMS instruments then will be turned on and allowed to warm up, and an initial response time test will be performed per the procedure and baseline environmental conditions of Section 6.1. A single TIC (AC) will be used in this evaluation. The indicated concentration signal from the IMS instruments will be recorded. At each sampling of the AC mixture, the instrument's battery level indication will be recorded. The instruments will then sample clean air for 30 min, then the AC mixture will be sampled again. This procedure will be repeated until the battery is exhausted, or until the IMS units no longer respond to the presence of AC. The total time of operation will be recorded as the measure of battery life.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

7.1 Equipment Calibrations

7.1.1 Reference Methods

The reference methods to be used for the determination of TICs and CW agents are listed in Section 4.7. The analytical equipment needed for these methods will be calibrated, maintained and operated according to the quality requirements of the respective methods or SOPs indicated in Section 4.7, and the normal operational procedures of the test facility. Continuous monitoring equipment, such as an electrochemical monitor used to confirm Cl_2 concentrations, will be operated, calibrated, and maintained according to the pertinent operations manual for the equipment.

7.1.2 IMS Instruments Checks

The IMS instruments will be operated and maintained according to the vendor's instructions throughout the verification test. Vendors will be required to provide such instructions before testing. Maintenance will be performed only according to a preset schedule or in response to predefined IMS instrument diagnostics.

7.2 Assessment and Audits

7.2.1 Technical Systems Audits

Battelle's Quality Manager will perform a TSA at least once during the performance of this verification test. The purpose of this TSA is to ensure that the verification test is being performed in accordance with this test/QA plan and that all QA/QC procedures are being implemented. In this audit, the Quality Manager may review the reference sampling and analysis

methods used, compare actual test procedures to those specified in this plan, and review data acquisition and handling procedures. The Quality Manager will prepare a TSA report, the findings of which must be addressed either by modifications of test procedures or by documentation in the test records and report.

At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to testing staff at the time of the audit, and documented in a TSA report.

7.2.2 Performance Evaluation Audit

A PE audit will be conducted to assess the quality of the measurements made in this verification test. This audit addresses only those reference measurements that factor into the data used for verification, i.e., the IMS detection technologies are not the subject of the PE audit. This audit will be performed once during the verification test, and will be performed by analyzing a standard that is independent of standards used during the testing. Table 7 summarizes the PE audit that will be done and indicates the acceptance criteria for the PE audit. This audit will be the responsibility of Battelle and test facility staff. As indicated by Table 7, the PE audit will be conducted for TICs, but not for the CW agents. The reason for this is that there is no independent source of the CW agents, i.e., all agents used in testing are obtained from the U.S. Army. In lieu of a PE audit for the CW agents, sorbent traps will be spiked with known quantities of the agents, and subjected to analysis as a QA check. This check will be conducted once in each verification test, with at least one spiked sorbent trap prepared for each of the two CW agents (GB and HD) used in testing. The target agreement for this QA check will be ± 30%. Also, no PE audit will be done for the TIC cyanogen chloride (CK) because the source gas for that TIC is prepared by Battelle, and no independent standard is available.

In the event that results of analysis of the PE audit standard do not meet the acceptance criteria, then the reference analysis method will be recalibrated with the laboratory standards, and then the PE audit standard will be reanalyzed. Continued failure to meet the PE audit criteria

will result in the pertinent data being flagged, and the purchase of new standards for repetition of the PE audit. Battelle's Quality Manager will assess PE audit results.

Table 7. Summary of PE Audit

Parameter	Audit Procedure	Expected Tolerance
TIC Concentrations[a]	Analyze independent standards	± 20%

a: AC, SA, CG, and Cl_2 only.

7.2.3 Data Quality Audit

Battelle's Quality Manager will audit at least 10 % of the verification data acquired in the verification test. The Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons, and to final reporting. All calculations performed on the data undergoing audit will be checked.

7.2.4 Assessment Reports

Each assessment and audit will be documented in accordance with Sections 3.3.4 (Internal Assessment Reporting) and 3.3.5 (Response) of the program QMP.[(1)] Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Space for response to adverse findings or potential problems
- Possible recommendations for resolving problems
- Citation of any noteworthy practices that may be of use to others
- Confirmation that solutions have been implemented and are effective.

Copies of the TSA assessment report will be provided to the EPA QA Manager.

7.2.5 Corrective Action

The Quality Manager during the course of any assessment or audit will identify to the technical staff performing experimental activities any immediate corrective actions that should be taken. If serious quality problems exist, the Quality Manager is authorized to stop work. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective actions. The Quality Manager will ensure that follow-up corrective actions have been taken.

8.0 DATA ANALYSIS AND REPORTING

8.1 Data Acquisition

Data acquisition in this verification test includes proper recording of the procedures used in testing, to assure consistency in testing and adherence to this plan; documentation of sampling conditions and analytical results for the reference methods; recording of the readings of the IMS instruments in each portion of the test; and recording of observations about ease of use, cost, etc. These forms of data acquisition will be carried out by the testing staff, in the form of laboratory record books, analytical data records, and data recording forms.

Table 8 summarizes the types of data to be recorded, how the data will be recorded, and how often the data will be recorded. All data will be recorded by Battelle staff. The general approach is to record all test information immediately and in a consistent format throughout all tests. This process of data recording and compiling will be overseen by the Battelle Verification Test Coordinator and Quality Manager.

8.1.1 IMS Data Acquisition

The acquisition of data from the IMS instruments will be tailored to the data output capabilities of those instruments. It is expected that a visual display of readings, coupled with an audible or visual alarm, will be the data output of most portable IMS instruments. For those IMS instruments, data will be recorded manually by the testing staff, on data forms prepared before the verification test. Separate forms will be prepared for distinct parts of the test, and each form will require entries that assure complete recording of all test data.

Some IMS instruments may have on-board data logging capabilities, or may provide an electronic output signal. In such cases, data acquisition will be conducted electronically, using the IMS instrument's own software or a personal computer-based data acquisition system in the test facility.

Table 8. Summary of Data Recording Process for the Verification Test

Data to be Recorded	Where Recorded	How Often Recorded	Disposition of Data[(a)]
Dates, times of test events	Laboratory record books, data forms	Start/end of test, and at each change of a test parameter.	Used to organize/check test results; manually incorporated in data spreadsheets as necessary.
Test parameters (agent/surrogate identities and concentrations, temperature and relative humidity, gas flows, etc.)	Laboratory record books, data forms	When set or changed, or as needed to document the sequence of tests.	Used to organize/check test results, manually incorporated in data spreadsheets as necessary.
Reference method sampling data (identification of sampling media, sampling flows, etc.)	Laboratory record books, data forms	At least at start/end of reference sample, and at each change of a test parameter.	Used to organize/check test results; manually incorporated in data spreadsheets as necessary.
Reference method sample analysis, chain of custody, and results	Laboratory record books, data sheets, or data acquisition system, as appropriate.	Throughout sample handling and analysis process	Transferred to spreadsheets
IMS instrument readings and diagnostic displays	Electronically if possible; prepared data forms otherwise	When stable at each new clean air, interferent, or challenge concentration; whenever updated in recovery and response time tests	Transferred to spreadsheets

(a) All activities subsequent to data recording are carried out by Battelle.

Whether collected manually or electronically, all IMS data will be entered into electronic spreadsheets, set up to organize the IMS, reference method, and test condition (e.g., temperature, RH, interferent concentration) data for each part of the test procedure. Organization of the data in this way will allow evaluation of the various performance parameters clearly and consistently. The accuracy of entering manually-recorded data into the spreadsheets will be checked at the time the data are entered, and a portion of the data will also be checked by the Battelle Quality

Manager as part of the Data Quality Audit (Section 7.2.3). A separate spreadsheet will be set up for each IMS instrument tested, and no intermingling or intercomparison of data from different instruments will take place.

8.1.2 Laboratory Data Acquisition

Laboratory analytical data (e.g., reference method results quantifying the TICs or CW agents used) may be produced electronically, from (e.g.) gas chromatographic or electrochemical instruments. For IMS instruments that do not provide an electronic output, data will be recorded manually in laboratory record books or on data forms prepared prior to the test. These records will be reviewed at least on a weekly basis to identify and resolve any inconsistencies. All written records must be in ink, and signed (or initialed) and dated by the person recording the information. All written records must be entered promptly, legibly, and accurately. Any corrections to notebook entries, or changes in recorded data, must be made with a single line through the original entry. The correction is then to be entered, initialed and dated by the person making the correction.

8.1.3 Confidentiality

In all cases, strict confidentiality of test data for each vendor's technology, and strict separation of data from different technologies, will be maintained. Separate files (including manual records, printouts, and electronic data files) will be kept for each technology. At no time during verification testing will Battelle staff engage in any comparison of different technologies undergoing testing.

8.2 Data Review

Records generated in the verification test will receive a one-over-one review within two weeks after generation, before these records are used to calculate, evaluate, or report verification results. These records will include laboratory record books, completed data forms, electronic spreadsheets or data files, and reference method analytical results. This review will be performed by the Battelle Verification Test Coordinator or his designate, but in any case someone other than the person who originally generated the record. Testing staff will be consulted as needed to clarify any issues about the data records. The review will be documented by the person performing the review by adding his/her initials and date to a hard copy of the record being reviewed.

8.3 Data Evaluation

In order to extract the most information about IMS performance from the test procedures, a multivariate statistical analysis of the test results will be performed whenever feasible. Such an analysis will use all available data to explore the impact of test parameters on IMS performance. However, a potential limitation in this approach is that some instruments to be tested under this test/QA plan may provide primarily qualitative responses. That is, they may indicate the presence or absence, and in some cases the relative concentration, of a target TIC or CW agent, rather than a quantitative concentration. As a result, for some IMS instruments the data produced in this test may not lend themselves to multivariate analysis. To address this limitation, a multivariate analysis is planned, but is backed up by single-variable analyses that will be employed as needed. Section 8.3.1 below describes the multivariate approach, and Section 8.3.2 describes the alternative single variable analyses.

8.3.1 Multivariate Analyses

The multivariate analyses focus on the following IMS detector performance parameters:

- Response Time
- Recovery Time
- Accuracy
- Repeatability
- False positives/False negatives,

by considering the following explanatory effects:

- Identity of the target TIC or CW agent
- Temperature
- Humidity
- Instrument Start State (i.e., warmed up, cold start, etc.)
- Identity and presence or absence of interferent

The performance parameters of response threshold and battery life do not lend themselves to a multivariate analysis based on the planned test procedures, and will be addressed using a single-variable approach (Sections 8.3.2.5 and 8.3.2.9).

8.3.1.1 Evaluation of Multiple Performance Parameters

For each IMS instrument, response and recovery time, accuracy, and repeatability will be measured with each target TIC and CW agent, at varying conditions of four environmental variables: temperature, humidity, start state, and interferent. At least five measures of the performance parameters will be taken for each combination of TIC/agent and environmental variables. Furthermore, since two units of each IMS instrument will be tested simultaneously, up to ten measures of each performance parameter will be available for each combination. Thus, for example, since three temperature levels will be assessed (5, 20 and 35 °C) at a fixed humidity (50% RH) and start state (warmed-up) – at least five measures of the performance parameters will be available for each TIC/CW agent and temperature combination.

A multivariate analysis of variance (MANOVA) will be performed to quantify IMS performance and to understand how IMS performance relates to TIC/CW agent identity and the values of the environmental variables. Given the experimental design, it is not anticipated that it will be possible to uncover interactions between temperature, humidity, and the other variables. For example, the design is limited to recording IMS response as temperature varies at one level of humidity, and recording IMS response as humidity varies at one level of temperature. For reasons of experimental practicality, the design does not include simultaneously high values of temperature and humidity. However, the data analysis will consider environmental interactions and the degree to which available data do in fact allow for their exploration.

8.3.1.2 False Positives and False Negatives

A representative set of potentially interfering compounds will be added to air samples, both with and without a target TIC or CW agent present in the samples. Some IMS instruments may provide only a binary (yes/no) response indicating the detection or non-detection of the target TIC/CW agent. At least five such binary responses will be collected for each interferent/zero air and interferent/TIC or agent combination. The false positive and negative rates of the IMS will be modeled in such cases using logistic regression, a technique that relates the chance of an event (for example, the chance of a positive reading when no TIC/CW agent is present) to explanatory variables (for example, interferent). The focus of the analyses will be to understand the relationship between false positive rate and interferent; and false negative rate and interferent/TIC or agent combination. For IMS instruments that provide a quantitative measure of the TIC or CW agent concentration, an analysis will be conducted to assess whether significant differences in response result from the presence of the interferent. Both types of analyses will use data from tests conducted with the interferent species, and corresponding data from other parts of the test procedures in which no interferent was present.

8.3.1.3 Support Tools

All data analyses will be conducted using the statistical analysis software, SAS. The SAS software provides extensive analytical capabilities, handling a wide range of statistical analyses,

including analysis of variance, regression, categorical data analysis, multivariate analysis, survival analysis, cluster analysis, and nonparametric analysis. As indicated, the analyses described above will rely primarily on SAS' support of multivariate analysis of variance and logistic regression. SAS tools will also be used for data summarization, including visualization of data with high-resolution graphics.

8.3.2 Single-Variable Analyses

8.3.2.1 Response Time

The data collected to evaluate response time will be the measured time periods (in seconds) between the start of IMS sampling from the challenge plenum and the achievement of an alarm state, or a switch to the backflush mode, on the challenge gas. These data will be recorded in sets of five, as a result of alternately sampling clean air and the challenge gas five successive times. Five replicate response time measurements will be recorded in all tests in which the IMS instruments are challenged with a test mixture, whether that mixture is of a TIC, a CW agent, or an interferent. The only exception is that if no effect is observed from an interferent after three replicates, the final two replicates will not be conducted.

The recorded response time data will be tabulated in the verification report, and will be summarized in terms of the mean and range of response times observed. Data analysis will include comparison of the observed means and ranges of response times under different test conditions. For example, response time may vary as a function of the target analyte concentration, so the response times will be compared graphically (linear regression) and/or statistically (comparison of means) to determine whether there is a significant dependence of response time on concentration. Linear regression analysis will focus on whether a statistically significant slope and correlation result from the regression of IMS results against reference method concentration data. Comparison of means will assess whether the mean response time at one concentration differs from that at another concentration. Corresponding comparisons will be made to assess the effect of temperature, RH, and the presence of interferents on response times.

These comparisons will be carried out using data for each TIC and CW agent tested, and consequently the response time will be assessed separately for each such target chemical.

8.3.2.2 Recovery Time

Recovery time will be evaluated in the same manner as described above for response time in Section 8.3.2.1, except that the data points will be the time from switching the IMS sampling point to the clean air plenum until baseline IMS response, the absence of an alarm, or a return from backflush mode is achieved. As is the case for response time, recovery time will be evaluated for all test runs, for all TICs and agents tested, by means of the mean and range of the values found in each test.

8.3.2.3 Accuracy

Accuracy will be assessed by comparing the IMS readings with the reference method results, for each TIC and agent tested. The comparison will be conducted differently for quantitative IMS results relative to qualitative results.

For IMS instruments that provide quantitative data, accuracy will be assessed by a linear regression of IMS data against reference method data. This comparison will be conducted separately for each TIC and agent tested, and will use all test results. Results from tests at the baseline conditions (22 °C and 50% RH) with no interferent present will be segregated from those at other test conditions, or with interferents present, but the same comparisons will be conducted on all data sets. The comparison will assess whether the slope of the regression line is significantly different from 1.0 and whether the intercept of the regression line is significantly different from zero.

For IMS instruments that provide qualitative data output, the assessment of accuracy will depend on information provided by each IMS vendor on the correspondence of qualitative readings to quantitative values. Accuracy will then be assessed by comparing the reference method data with the ranges of concentration indicated by qualitative IMS readings. This comparison will result in a Yes/No (Y/N) assessment of accuracy for each reference/IMS data set. For example, an IMS vendor whose instrument provides a low/medium/high indication

reports that the "medium" response range for a particular chemical agent corresponds to concentrations of 5 to 10 (arbitrary units for example only). Then any IMS reading of "medium" that corresponds with a reference method result of 5 to 10 units will be designated as accurate (Yes); "medium" readings that correspond to reference values outside the 5 to 10 range will be designated as inaccurate (No). The results will be tabulated and the Y/N results will be reviewed. As with the quantitative data, qualitative accuracy will be assessed for each TIC and agent, using all test data.

For IMS instruments that provide only an alarm, or that switch into a backflush mode and stop sampling upon detection of the target species, accuracy will be assessed only in terms of false positives and false negatives. For this evaluation, a positive IMS response in the absence of the TIC or CW agent concentration will be deemed a false positive, and the absence of IMS response at any concentration above the response threshold for the target species will be deemed a false negative.

8.3.2.4 Repeatability

Repeatability will be assessed by means of the stable IMS readings recorded in the successive periods of sampling from the challenge plenum, at each concentration of TIC or CW agent. Each set of five replicate readings will be tabulated, and the consistency of readings will be noted as a function of the identity and concentration of the target analyte, the temperature and RH, and the presence of an interferent. In the case of IMS instruments that provide only alarms or qualitative responses, the evaluation of repeatability will be necessarily qualitative. That evaluation will be conducted by noting, for example, whether all three readings in a test run were the same, or two out of three were the same, etc. The exact nature of this qualitative evaluation will depend on the nature of the data output provided by the IMS instrument. In the simplest form, the evaluation of repeatability may involve only the consistency of providing an alarm or switching into a backflush when the TIC or agent is present.

For IMS instruments that provide a quantitative data output, repeatability will be assessed in terms of the percent relative standard deviation (%RSD) of the five readings from each test, i.e.,

$$\%RSD = (SD/Mean) \times 100$$

where SD is the standard deviation of the five readings in a test, and Mean is the arithmetic average of the five readings.

The %RSD results will be evaluated by inspection, and apparent differences in repeatability will be tested for significance by a comparison of means test (Student's t or similar).

8.3.2.5 Response Threshold

The data used to evaluate the response threshold will be the five replicate IMS readings obtained at each succeeding target analyte concentration, in the procedure described in Section 6.2. These data will be tabulated, along with the corresponding reference method data that establish the challenge concentration. The response threshold will be determined by inspection as the lowest reference method concentration that produces a positive IMS response in all triplicate runs. In this evaluation, the consistency of the IMS readings is not an issue, e.g., an IMS response of "low" is equivalent to a response of "medium" or "high" in terms of the response threshold evaluation.

8.3.2.6 Temperature and Humidity Effects

Temperature and humidity effects will be assessed by direct comparison of test results under baseline conditions (22°C and 50% RH) to those under other conditions. Temperature or RH effects will be examined relative to each of the performance parameters being tested, i.e., response time, recovery time, accuracy, etc. Thus assessment of temperature or RH effects involves comparison of results for those performance parameters under different temperature and RH conditions.

These effects will be evaluated by tabulation of the results obtained for the various performance parameters, under each set of temperature and RH conditions. Identification of temperature or RH effects will begin by inspecting the data for apparent differences that may be

a function of temperature or RH. Any suspected differences will then be investigated by appropriate means, such as linear regression or comparison of means. The effect of temperature will be assessed by comparing data from the tests conducted at 10 to 35°C with constant 50 (±5) % RH; the effect of RH will be assessed by comparing data from the tests at ≤ 20 to 80% RH at constant 22 (±3) °C temperature. These evaluations will be done separately for each TIC and CW agent tested.

8.3.2.7 Interference Effects

The impact of interferences on IMS response will be assessed by comparison of response with a potential interferent present to that in the absence of interferent, under the same test conditions. Response will consist of the readings of the IMS instrument in tests both with and without the interferent. Comparison of these responses may conveniently be done graphically, to illustrate the difference or similarity of the responses. All response readings with the interferent present must be the same as those without the interferent present, or an interferent effect will be inferred. For example, three positive and two negative responses in the presence of the interferent will be judged as different from two positive and three negative responses in the absence of the interferent indications.

The interference data will be evaluated in two ways. Data from the tests with interferent present alone will be used to assess false positive readings, i.e., comparison of IMS readings with interferent and clean air will assess whether the IMS instruments give a positive indication of a TIC or agent due to the presence of interferent. Data from the tests with both interferent and a TIC or agent will be used to assess false negatives, i.e., the absence of a response to the TIC or agent when the interferent is present. A reduced or enhanced response to the TIC or agent when the interferent is present, relative to that without the interferent, will be taken as indication of a partial masking or interference in the IMS response.

This evaluation will be conducted by matching (in the data spreadsheets) the results from tests with interferents present with those at the same conditions without interferents. This organization of the data will be done separately for each TIC or CW agent tested, so that interferent effects are assessed separately for each TIC or agent.

8.3.2.8 Cold/Hot Start Behavior

One evaluation of cold/hot start behavior will use the measured time between the startup of the IMS instrument and when it is ready to provide data. Three values of this result will be tabulated: one resulting from a cold start from room temperature, another from a cold start after prolonged storage at reduced temperature (5 to 8°C), and the third from a cold start after prolonged storage in a hot environment (40ºC). These three measured delay times will be reported without any additional data analysis.

Additional evaluation of cold/hot start behavior will result from the determination of response time, repeatability, and recovery time in the tests that immediately follow the cold and hot starts. These data, which will result from the determination of these performance parameters as described elsewhere in Section 8.3, will be compared to those from tests under the same baseline conditions with full warmup prior to testing. Differences in performance between cold/hot start and warmed up operation will be investigated by comparing the mean values and ranges of the results.

8.3.2.9 Battery Life

Both battery life and the effectiveness of battery operation will be assessed. Battery life will be reported as the time (in minutes) from startup to battery exhaustion when an IMS instrument is warmed up and operated solely on battery power at room temperature and 50% RH. This time will be measured from initial startup of the instrument to the point in time when the IMS instrument ceases to function or no longer responds to a challenge mixture of a selected TIC in air.

If feasible for the IMS instruments, the effectiveness of battery operation will be assessed by comparing the triplicate test results for a single TIC with the IMS instrument operated on AC power, to the corresponding results when the same test is immediately repeated using IMS battery power. The results for response time, recovery time, accuracy, and repeatability will be compared to assess whether any substantial differences result from use of battery power.

8.4 Reporting

The data comparisons described in Section 8.3 will be conducted separately for each IMS instrument undergoing verification. Separate verification reports will then be prepared, each addressing one IMS technology. Each verification report will present the test data, as well as the results of the evaluation of those data. The verification report will briefly describe the ETV program, and will present the procedures used in verification testing. These sections will be common to each verification report resulting from this verification test. The results of the verification test will then be stated quantitatively, without comparison to any other technology tested, or comment on the acceptability of the technology's performance. The preparation of draft verification reports, the review of reports by vendors and others, the revision of the reports, final approval, and the distribution of the reports, will be conducted as stated in the program QMP.[(1)] Preparation, approval, and use of Verification Statements summarizing the results of this test also will be subject to the requirements of that same document.

9.0 HEALTH AND SAFETY

All participants in this verification test (i.e., Battelle, EPA, and vendor staff) will adhere to the security, health, and safety requirements of the Battelle facility in which testing will be performed. Vendor staff will train Battelle testing staff in the use of their portable IMS instruments, but will not be the technology users during the testing. To the extent allowed by the test facility, vendor staff may observe, but may not conduct, any of the verification testing activities identified in this test/QA plan.

9.1 Access

Access to restricted areas of the test facility will be limited to staff who have met all the necessary training and security requirements. The existing access restrictions of the test facility will be followed, i.e., no departure from standard procedures will be needed for this test.

9.2 Potential Hazards

This verification in part involves the use of extremely hazardous chemical materials. Verification testing involving those materials must be implemented only in properly certified surety facilities, capable of handling such materials safely.

In addition, TIC materials used in this verification may be toxic, and must be used with appropriate attention to good laboratory safety practices.

9.3 Training

Because of the hazardous materials involved in this verification test, documentation of proper training and certification of the test personnel is mandatory before testing takes place. The Battelle Quality Manager, or a designate, must assure that documentation of such training is in place for all test personnel before allowing testing to proceed.

9.4 Safe Work Practices

All visiting staff at the test facility will be given a site-specific safety briefing prior to the start of any test activities. This briefing will include a description of emergency operating procedures, and the identification and location and operation of safety equipment (e.g., fire alarms, fire extinguishers, eye washes, exits). Testing procedures must follow all safety practices of the test facility at all times. Any report of unsafe practices in this test, by those involved in the test or by other observers, shall be grounds for stopping the test until the Quality Manager and testing personnel are satisfied that unsafe practices have been corrected.

9.5 Equipment Disposition

Tests conducted according to this plan will require that all equipment that has been exposed to chemical surety materiel be decontaminated and/or disposed of. Although efforts will be made to remove any contaminated parts of the IMS instruments after testing, there is no guarantee that this will be feasible. Consequently, it is not certain that IMS instruments undergoing testing will be returned to the vendor at the completion of the tests.

10.0 REFERENCES

1. Quality Management Plan (QMP) for the ETV Safe Buildings Monitoring and Detection Technology Verification Program, Version 1, Battelle, Columbus, Ohio, June 1, 2004.

2. Battelle Gas Chromatography Method for Cyanogen Chloride (CK), Method Designation C:\HPCHEM\1\METHODS\ETV_CK.M, May 2003.

3. Battelle Chromatography Method for Hydrogen Cyanide (HCN), Method Designation C:\HPCHEM\1\METHODS\ETV_HCN.M, May 2003.

4. "Determination of Phosgene in Air", in Methods of Air Sampling and Analysis, Third Edition, J. P. Lodge ed., Lewis Publishers, Chelsea, Michigan, 1989.

5. Battelle Gas Chromatography Method for Arsine, Method Designation C:\MSDCHEM\1\METHODS\OLD\ARSINE5.M, May 2004.

6. Proposed Acute Exposure Guideline Levels (AEGLs), Nerve Agents GA, GB, GD, GF, U.S. EPA, Office of Pollution Prevention and Toxics, Public Draft, October 2000. Federal Register (www.access.gpo.gov/su_docs/aces/aces140.html).